



PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Tatsuya MATSUI et al.

Group Art Unit: 1711

Application No.: 10/518,522

Examiner: S. ACQUAH

Filed: April 4, 2005

Docket No.: 122137

For: ADDITIVES FOR CEMENT

DECLARATION UNDER 37 C.F.R. §1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Tatsuya MATSUI, a citizen of Japan, hereby declare and state:

1. I have a Master of Science degree, which was conferred upon me by Yokohama National University in 1996.
2. I have been employed by NOF CORPORATION since 1996 and I have had a total of ten years of work and research experience in specialty chemical research. I currently hold the position of Research Member of the Oleo & Speciality Chemicals Research Laboratory.
3. I am a named inventor in the above-captioned patent application.
4. I have a professional relationship with the assignee of the above-identified patent application. In the course of that professional relationship, I received compensation directly from the assignee for my work relating to cement additives. I am being compensated for my work in connection with this Declaration.

5. I and/or those under my direct supervision and control have conducted the following tests:

This experiment was carried out according to the disclosure of "manufacturing example 3" in paragraphs (0008) to (0013) of JP 06-298556A and page 31, lines 2 to 20 of the specification of U. S. Patent Application Serial No. 10/518,522.

A. Production of the Copolymer "B" of JP 06-298556A

64 grams of methanol and 2.0 grams of sodium methoxide were charged into a 5 liter pressurized reaction vessel. Air in the vessel was replaced with nitrogen, and 2904 grams of ethylene oxide was gradually added at a pressure of 0.05 to 0.5 MPa (gauge pressure) at 100 to 120 °C to perform addition reaction. The reaction mixture was then cooled to 50 °C after the completion of the reaction.

112 grams of potassium hydroxide was then added and air was replaced with nitrogen. 153 grams of allyl chloride was then gradually added to the reaction mixture while agitating at 80 °C. The reaction was terminated after 6-hour agitation.

The reaction mixture was neutralized with hydrochloric acid and the by-product (salt) was removed to obtain a polyoxyalkylene compound.

1524 grams (1 mole) of the polyoxyalkylene compound, 98 grams (1 mole) of maleic anhydride and 300 grams of toluene were charged into a 3-liter flask equipped with an agitator, a thermometer, a tube for introducing nitrogen gas, a dropping funnel and a condensor.

13.1 grams of 2,2-azobisisobutyronitrile was dissolved into 262 grams of toluene and dropped into the flask at 85 °C \pm 2 °C for 3 hours under nitrogen gas atmosphere. After the completion of the dropping, the reaction was further continued for 3 hours at 85 °C \pm 2 °C. Toluene was removed under reducing atmosphere to obtain the copolymer "B".

B. Production of the Esterified Product According to the Manufacturing

Example 3 of JP 06-298556A

877 grams of the copolymer "B", 123 grams of $(\text{CH}_3)_2\text{N}(\text{C}_2\text{H}_4\text{O})_{11}\text{H}$ and sodium methylete were charged into a two-liter and 4 neck flask equipped with an agitator, a tube for blowing nitrogen, a thermometer and a condenser. The amount of sodium methylete was 0.1 weight percent with respect to that of the copolymer "B". The mixture was reacted for 3 hours at 100 °C to obtain the esterified product.

C. Concrete Test

The solution of the esterified product obtained in Section B. above was diluted with ion exchange water to adjust the content of aqueous solution at 20 weight percent, to which an anti foaming agent ("DISFOAM CC-118" supplied by NOF CORPORATION) was added.

Concrete was prepared in a laboratory at 30 °C as follows.

10.9 kg of cement (ordinary portland cement), 26.0 kg of fine aggregates (river sands from Ooi river; specific gravity of 2.60) and 28.9 kg of coarse aggregates (crushed stones from Oume: specific gravity of 2.66) were supplied into a 50-liter forced biaxial mixing type mixer to perform dry mixing for 15 seconds. After that, the above aqueous solution of the esterified product was added to the mixture in an amount of 196 grams at 30 °C with 4.4 kg of civil water and mixed for 2 minutes. The amount was controlled so that the slump of the composition direct after the mixing is 20 ± 1 cm.

The composition was discharged to a vat for retempering to measure the slumps of the composition direct, 30 minutes, 60 minutes and 90 minutes after the mixing. It was confirmed that air content was 4.5 ± 1.0 % and the temperature was 30 ± 2 °C between time points direct after and 90 minutes after the mixing.

The results are shown in the following table. Column of "added amount" indicates that as 20 % aqueous solution.

Ambient temperature	30 °C				
	Added amount C x %	Slump (cm)			
		Direct after mixing	30 minutes	60 minutes	90 minutes
The esterified product of manufacturing example 3	1.80	20.0	20.5	18.0	15.0

6. I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Date: August 22, 2006

Tatsuya Matsui
Tatsuya MATSUI